

In still another embodiment of the present invention the process ensures better control of RE concentration in the doped region and homogeneous distribution of RE ions along the radial direction as well as throughout the length of the preform.

In yet another embodiment of the present invention the RE incorporation efficiency is much higher compared to the conventional techniques because of direct addition of the RE oxides into the dispersion instead of the corresponding salt by the conventional techniques thereby minimising the possibility of evaporation and change in concentration.

In still another embodiment of the present invention the addition of  $\text{Ge}(\text{OC}_2\text{H}_5)_4$  at ambient temperature in the silica sol above reduces the quantity of  $\text{GeCl}_4$  which is required at high temperature to achieve the desired NA.

In yet another embodiment of the present invention the time period of processing the silica tube at high temperature and the number of steps involved for doping of RE ions by the conventional techniques for fabrication of the preform are considerably reduced.

In still another embodiment of the present invention the processing of the tube at ambient temperature before sintering and collapsing instead of high temperature involved in the CVD process makes the process less sensitive to the process parameters unlike the conventional processes.

In still another embodiment of the present invention the advantages described above increases the reproducibility and reliability of the process to a great extent.

In yet another embodiment of the present invention the requirement of precision equipments for control of porous soot deposition, RE incorporation etc. during fabrication of the preform is considerably eliminated which will reduce the capital investment and cost of the product.

In still another embodiment of the present invention the advantages combined make the process simple and more economic than the conventional processes.

#### **Brief description of the drawings**

**Figure 1 & 2** represent respectively the spectral attenuation curve of Er doped fibre prepared from RE doped nanoparticles following the present process and that fabricated by conventional solution doping technique.

**Figure 3 & 4** show the refractive index profiles of Er doped fibre prepared through nanoparticle route and that by solution doping method respectively.

The invention is further explained with the help of following examples which should not be construed to limit the scope of the invention:

#### Sonochemical preparation of $\text{RE}_2\text{O}_3$ - coated silica nanoparticles

following the process mentioned in the Ref: ‘Sonochemical Preparation and Characterization of  $\text{Eu}_2\text{O}_3$  and  $\text{Tb}_2\text{O}_3$  Doped in and Coated on Silica and Alumina Nanoparticles’ A.Patra, E. Sominska, S. Ramesh, Yu. Koltypin, Z. Zhong, H. Minti, R. Reisfeld and A. Gedanken, J. Phys. Chem. B, Vol 103 (17) pp 3361-3365.

(It does not form a part of the invention )

Amorphous silica microspheres in the size range of 50-250 nm were synthesized by the alkaline hydrolysis of tetraethoxysilane (Stober method). For the preparation of the  $\text{RE}_2\text{O}_3$ - coated silica nanoparticles with molar composition  $(100-x)\text{SiO}_2-x\text{RE}_2\text{O}_3$ , nanophased rare earth oxides were sonochemically deposited on the outer surface of spherical silica particles. For this purpose, rare earth nitrate used as the source of rare earth was prepared by dissolving rare earth oxide in a minimum amount of nitric acid followed by evaporating it to dryness. The dry nitrate was dissolved in calculated quantity of water to prepare the rare earth nitrate solution.

The required amount of silica microspheres was taken in a beaker and calculated quantity of water and rare earth nitrate solution, as prepared earlier, were then added to it. The open beaker with the material was kept in an ice-bath and subjected to sonication for 1h employing a direct immersion titanium horn (Vibracell, 20 kHz, 100 W/cm<sup>2</sup>). Required amount of 25% aqueous ammonia was thereafter added in drops into the beaker during sonication. The resulting product after sonication was washed thoroughly with water, centrifuged and finally dried under vacuum to obtain RE coated amorphous silica nanoparticles.

The above method is also suitable for the preparation of doped and co-doped silica particles containing  $\text{Al}_2\text{O}_3$ ,  $\text{GeO}_2$ ,  $\text{Yb}_2\text{O}_3$  and other rare earth oxides.

## EXAMPLE 1

### **Er-doped fibre**

- Amorphous silica microspheres synthesized by hydrolysis of tetraethoxyorthosilicate (Stober method) were dispersed in a solution of erbium nitrate (kept in an ice bath) in a proportion of 98.5 mol%  $\text{SiO}_2$  and 1.5 mol%  $\text{Er}_2\text{O}_3$  under sonication followed by the addition of aqueous ammonia by known process. The resulting product was washed with water followed by centrifugation and drying under vacuum.
- A stable dispersion of composition 94.98 $\text{SiO}_2$ :3 $\text{GeO}_2$ :2 $\text{Al}_2\text{O}_3$ : 0.02 $\text{Er}_2\text{O}_3$  (in equivalent oxide mol%) was prepared for the application of coating to the inner wall of high purity clear fused silica glass tubes.
- From the erbium oxide ( $\text{Er}_2\text{O}_3$ ) coated silica powders with 98.5 mol%  $\text{SiO}_2$  and 1.5 mol%  $\text{Er}_2\text{O}_3$ , a silica sol of composition of 94.98 equivalent mol% of  $\text{SiO}_2$  and 0.02 equivalent mol% of  $\text{Er}_2\text{O}_3$  was prepared by diluting with a silica sol containing the desired amount of silicon tetraethoxide (TEOS).
- Silica-germania sol containing 3 equivalent oxide mol% of germanium ethoxide  $[\text{Ge}(\text{OC}_2\text{H}_5)_4]$  was prepared through the hydrolysis of TEOS and  $[\text{Ge}(\text{OC}_2\text{H}_5)_4]$  with water and hydrochloric acid in presence of a mixed solvent of propan-1-ol and butan-2-ol. pH of the above sol was  $1.5 \pm 0.05$ .
- 2 equivalent oxide mol% of  $[\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}]$  and 0.02 equivalent mol% of  $\text{Er}_2\text{O}_3$  through  $\text{Er}_2\text{O}_3$  coated  $\text{SiO}_2$  powders (after baking at  $100^\circ\text{C}$  for 1h) were dispersed in the above silica sol under sonication (26 kHz) for 80 mins.
- The resultant dispersion after allowing to settle for 2 h, was used to coat the inner wall of the thoroughly cleaned silica glass tubes with inner diameter of 17.8 mm. The outer wall of the tubes were properly masked with a suitable substance (parafilm).
- Coating was performed by dipping the silica glass tubes with a speed of 8 cm/min into the dispersion and lifting the same tube from the above dispersions with the same speed.
- The coated tubes were dried in air at  $100^\circ\text{C}$  for 1h.